Rentran Translation Services
Gerd Renno
Rt. 2, Box 86; Deer Creek Road
Weatherford, OK 73096
(o) (580) 774-3858
Fax: (580) 774-3413

3M Language Society Translation # 0584: **Patent Disclosure DE 196 49 728 A1**

Intern. Class: C 09 J 7/02

German Patent Office

Patent Disclosure
DE 196 49 728 A1

File number: 196 49 728.0
Date of application: November 11, 1996
Date of disclosure: June 18, 1998

Applicant: Beiersdorf AG, 20253 Hamburg, DE

Inventor: Lühmann, Bernd, Dr., 22846 Norderstedt, DE;
Junghans, Andreas, 22457 Hamburg, DE

Oppositions: DE 44 28 587 C2
WO 95 06 691 A1

Following statements are taken from the files submitted by the applicant

Adhesive Tape

Adhesive tape for an adhesive joint which can be released by stripping residue free and without damage, with a foam backing, which is coated on one or both sides with a self-adhering adhesive material, characterized by

a) having a self-adhering adhesive material applied to at least one of the two sides of the foam backing, whose ratio of rupture force to strip force (removal force) at a strip angle of less than 10° to the adhesive area is larger than 1.2 : 1, and
b) the foam backing featuring an elasticity modulus below 16 MPa and a thickness of 200 - 600 µm.

Description

The invention concerns an adhesive tape for an adhesive joint which can be removed by pulling residue free and without damage, and its application.

Highly extensible, elastic adhesive films for removable adhesive joints which can be removed largely by pulling in the direction of the adhesive plain are well known and commercially available under the name "tesa Power-Strip". Joints produced with them offer a strong mount and can still be removed without residue and without damage to the backing or the parts to be joined as described in DE 33 31 016 C2. DE 42 22 849, DE 42 33 872, DE 44 28 587, DE 44 31 914, and DE 195 11 288 describe among other items a specific design and application of above specified adhesive films.

Multiple layer adhesive films which contain highly extensible, low elasticity, or also highly extensible, elastic film backings as well as the application of said adhesive films are also known, for example, from US 4,024,312 "Pressure sensitive adhesive tape for medical use having an extensible, elastic block copolymer backing", WO 92/11332 "Removable adhesive tape" (PSA tape using highly extensible backing with photopolymerized acrylic PSA), WO 92/11333 (PSA tape using highly extensible essentially inelastic backing), WO 93/01979 "Securing of stacks with stretch adhesive tape", and WO 94/21157 "Article support using stretch releasing adhesive".

For instance, WO 92/11333 describes an adhesive tape which can be released by pulling in the plain of the adhesive joint, which utilizes as backing a highly extensible, mainly non-resilient (not rubber elastic) film, which exhibits a retraction capacity of $< 50\%$ after elongation. US 4,024,312 describes PSA tapes which can be released similarly and are of a rubber-elastic nature.

It has been shown in practical applications that in general a high adhesive strength can be achieved with the above named self-adhering adhesive tapes on smooth and firm substrates. The adhesive strength is insufficient for numerous applications on rough substrates, especially for products of small thickness, but also for PSA tapes with higher layer thickness. The reason for the insufficient adhesive strength is probably due mainly because of an inadequate area of adhesion due to inadequate conformity of the adhesive tapes to rough and irregular shaped surfaces, especially if two rough surfaces have to be glued together. For example, often times adhesive areas of about 10% to 40% of the tacky surface are only achieved at practical application pressures (100 N/7.4 cm^2) in case of adhesive joints of planar materials with "tesa" power strips on painted, rough fiber wallpaper. However, the reason for inadequate adhesive joint strength for adhesive joints of smooth, straight surfaces can also come from an insufficient adhesive joint surface area. The cause may be the inclusion of air bubbles into the adhesive joint areas. Air bubbles like that can often times also not be completely eliminated by high contact pressure. Adhesive joints, which exhibit these kind of air inclusions, can in unfavorable cases have drastically reduced adhesion strength compared to samples glued over the whole area

which are free of bubbles.

US 5,516,581 and WO 95/066691 describe self-adhering tapes removable by stretching mainly in the plain of the adhesive joint, whose backing contain polymeric foams. In particular, WO 95/06691 describes self-adhering adhesive tapes releasing by stretching which are obtained from backing materials utilizing polymer foams, which feature a significantly improved compliance to rough and irregular shaped surfaces. High adhesive strengths can be realized with these products even on rough and irregularly shaped substrates due to the larger adhesive surface area which is being achieved.

The foam backings described in US 5,516,581 and WO 95/066691, however, exhibit a number of serious disadvantages:

- Only those kind of foam backings are utilized which do not rupture when the adhesive tape is released. Brake resistant backings of this kind, however, require in part a very complex multi-layer structure; see claims 13, 14, 17 in US 5,516,581 as well as claims 4, 15 in WO 95/066691.
- Single layer foam backings can only be utilized to a limited extend.
- Single layer foam backings according to Claim 1 from WO 95/06691 have to have a minimum thickness of at least 30 mils = 760 μm.
- Single layer foam backings according to Claim 1 from WO 95/06691 are further limited in that only those with an elasticity modulus (Young's modulus) of about < 2400 psi = 16.9 MPa are exclusively suitable.
- The retraction capacity of the adhesive tapes described in US 5,516,581 across the board is at < 50% after the release process; adhesive tapes with a noticeable elastic retraction capability can therefore not be utilized according to the invention.

A significant portion of the thin layer polymer foams which are commercially available is therefore excluded from the above mentioned applications.

The task of the presented invention was to overcome the above mentioned disadvantages. This is achieved by adhesive tapes as characterized in more detail in the claims, especially by

- Use of polymer foams as backing materials, which are characterized by an elasticity modulus of less than 16 MPa and a layer thickness of about 200 to about 600 μm, preferably between 300 and 550 μm,
- which in combination with adhesive materials which possess a high degree of stretching at high rupture strength at the same time, whereby
- the resulting self-adhering tapes after stretching, especially in the plain of the adhesion joint can be released residue free, and whereby neither the adhesive material nor the used foam backing will break during the release process.

Only the combination of highly resistant, highly stretchable adhesive materials with the previously mentioned polymer foams, which do not break during the release process, in combination with the adhesive materials used according to the invention, make it possible to realize very thin layer and still highly compliant self-adhering adhesive tapes, which can be removed residue free and free of damage by stretching, especially in the plain of the adhesive joint. Very large adhesion surface areas are achieved in comparison to adhesive films which do not utilize an intermediate foam backing because of the high compliance to rough substrates such as painted, rough fiber wallpaper, for example.

The use of appropriate, thin layer foam materials allows at the same time to achieve low release forces (strip forces), a significant advantage in practical applications. Lower strip forces furthermore allow for a destructive free release on even very sensitive backgrounds such as many wallpapers, for example, besides the ease in handling of the adhesive film during the release process.

An advantage of the self-adhering adhesive tapes of this invention is further that the structure of the rough background does show much less in the region of the adhesive joint for joints of thin, flexible materials on very rough backgrounds, for example when gluing thin papered posters on rough fiber wallpaper.

Finally, other advantages are encompassed by the small amount of material usage to realize adhesive tapes containing foam materials as intermediate backing layer, which can be removed without residue and destruction free by stretching especially in the plain of the adhesive joint.

Examples of Applications

Residue and destruction free, removable PSA tapes for:

Original closure applications, tacky adhesive on single or double side,
mounting of posters, pictures, calendars, post cards, signs, tacky hooks, also pre-fabricated;
labels, for example for pricing,
in general to join materials which have to be separated at a later point in time.

Materials used

Adhesive Materials

Used as tacky adhesive materials are preferably those on the basis of block-copolymers containing polymer blocks made up of vinyl-aromates (A-blocks), preferably styrene, and

those made up by polymerization of 1,3-dienes (D-blocks), preferably butadiene and isoprene. Based on experience both homo-polymer blocks as well as copolymer blocks can be utilized. Resulting block-copolymers may contain the same or different D-blocks, which are partially, selectively, or completely hydrated. Block-copolymers can exhibit a linear A-D-A structure. Block-copolymers with a radial shape as well as a star shape and linear multiple block-copolymers can also be utilized. Additional components can be A-D dual block-copolymers which may be present. Block-copolymers can be modified, for example, made functional by conversion with maleic acid anhydride. Block-copolymers of vinyl aromates and isobutylene can also be utilized based on this invention. All of the above named polymers can be used alone or as mixtures with each other. Typical use concentrations for the styrene block-copolymers are in the range between 15 %w and 75 %w, preferably in the range between 30 %w and 60 %w, especially preferably in the range between 35 %w and 55 %w.

Suitable as tackifiers are among others: colophony (pine tree resin) and its derivatives, aliphatic, aromatic-modified aliphatic, aromatic, and phenol-modified tacky resins, to name a few. Use concentrations of the resins are typically in the range of 15 %w and 75 %w, preferably in the range between 30 %w and 65 %w, especially preferably in the range between 35 %w and 60 %w. Ester of partially and fully hydrated colophony resin are preferably used when utilizing colophony and its derivatives.

Homo-polymers and copolymers of vinyl-aromates such as styrene or α-methyl-styrene, polyphenylene oxide, but also resins modified with phenylene oxide can be used as resins compatible with terminal blocks (mainly resins compatible with vinyl-aromatic blocks).

Other optimal mixture components encompass softener oils and liquid resins (use concentrations between 0 and a maximum of about 35 %w), filler materials (reinforced and non-reinforced). For example, silicone dioxide, especially synthetic silica, glass (milled or in form of beads), aluminum oxide, zinc oxide, calcium carbonate, titanium dioxide, anti-ozone agents, metal deactivators, etc.). Mixture components also include polymers which influence in particular the ozone resistance of block-copolymers such as polyvinylacetate and ethylene-vinylacetate copolymers, for example.

Of course, natural and synthetic polymers, for example natural rubber, synthetic polyisoprene, polybutadiene, polychloroprene, SBR, Kraton Liquid (Shell Chemicals), low molecular styrene-diene block-copolymers like Kraton LVSI 101 for example, polyisobutylene, etc. can be present as additional polymers, which may replace the block-copolymers containing vinyl-aromatics up to a content of about 50 %w.

Adhesive materials according to the invention can be cross-linked chemically, (for example by UV radiation, γ-radiation, or by radiation with fast electrons).

Adhesive materials according to this invention or optionally those whose tackiness is created only after thermal activation.

Suitable adhesive materials besides those described above on the basis of block-copolymer containing vinyl-aromatic components are those which possess a rupture strength and cohesion sufficient for the release process.

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Adhesive materials like that can be utilized by themselves or in combination with those on the basis of block-copolymers containing vinyl-aromatic components. Suitable according to the invention are tacky acrylate copolymers copolymerized with macromonomers, for example, whereby the macromonomers feature a glass transition temperature of > +40 C°. The high rupture strength of corresponding copolymers is probably being achieved by the association of the macromonomers. Suitable macromonomers are methacryloyl terminated polymethylmethacrylate, for example.

Foam Materials

Suitable foam materials (foam backings) are preferably based on polyolefine homo- and copolymers. Homo- and copolymers can be utilized by themselves or in mixtures. Especially preferred are ethylene-vinyl-acetate copolymers as well as mixtures of ethylene-vinyl-acetate copolymers and / or polyvinylacetates with polyethylene, whereby those polyethylens are preferably used which have low densities (LDPE, LLDPE, VLDPE). Suitable as additional polymers are: polyvinylacetate, EPDM, thermoplastic elastomers on the basis of styrene block-copolymers, polyurethane on the basis of aromatic and aliphatic diisocyanates, PVC, polychloroprene, and natural rubber.

The thicknesses of the foam materials utilized range, in particular, between about 150 µm and 600 m, preferably between 250 µm and 55 µm. Volume densities amount to about 40 kg/m^3 to 450 kg/m^3, preferably 50 kg/m^3 to 300 kg/m^3. Foams with or without skins can be used with integral or non-integral structure.

Anchoring of the Adhesive Material on Foams

In order to obtain adequate anchoring of the utilized adhesive materials on the foam material, the latter preferably undergo a pressure pretreatment during the foam production and / or before coating. Suitable pretreatment processes are among others a fluorine pretreatment, corona pretreatment, plasma pretreatment, and flame pretreatment; the latter especially by means of an electrically polarized flame, and the foam can be primed to additionally improve the adhesive anchoring in case of integrated foams.

An optional barrier layer can be integrated between the foam material and the adhesive material to reduce the migration of materials which are able to migrate between the

adhesive material and the backing.

Tacky Adhesive Tapes

Tacky adhesive tapes according to the invention contain at least a foam backing which is equipped on one or both sides with an adhesive material. The adhesive tapes possess an adequate rupture elongation and rupture strength, so that they can be removed residue free and non-destructive from the adhesion background by stretching, especially in the plain of the adhesive joint. The adhesive materials can be of the same or a different recipe in case of foam backings coated on both sides with adhesive material, and fully or partially covering the backing, for instance, on a striped line, as well as be coated at the same or a different coating thickness on both sides of the adhesive tape. Adhesive materials can consist of one or several adhesive material layers. For example, the adhesive material layer can consist of two layers which both utilize block-copolymers containing vinyl-aromatic components, whereby the block-copolymers are of a different kind. Also, a second layer on the basis of natural rubber or polybutadiene or polyisobutylene, or "Kraton Liquid" (Shell Chemicals), or mixtures of the previously listed polymers can be applied to a first adhesive layer of block-copolymers containing vinyl-aromatic components.

Tacky adhesive tapes are characterized by their rupture elongation being larger than 200%, preferably larger than 300%, especially preferably larger than 400%.

Rupture strengths of the adhesive materials used are especially larger than 1.5 MPa, preferably larger than 3 MPa, and especially preferably larger than 5 MPa.

Layer thicknesses of the adhesive materials determining the rupture strength reach to about ≥ 60 µm, preferably ≥ 100 µm, and especially preferably ≥ 150 µm.

The ratio of rupture strength to strip force of the tacky adhesive tapes at a strip angle of less than 10° to the adhesive plain is larger than 1.2 : 1, preferably larger than 1.5 : 1, and especially preferably larger than 2 : 1.

Packaging Forms

Packaging forms of the adhesive tapes of this invention encompass both adhesive tape rolls as well as tape pieces of defined dimensions, for example, in form of punched pieces. According to DE 44 28 587 adhesive tape pieces of defined dimensions can feature a ready-formed corner, for instance, an acute angle corner, or be equipped according to DE 44 31 914 with a strip film coated with a release layer or release layer coated release paper.

Production

Adhesive films of this invention can be produced by solvent coating, cold or hot lamination, and by melt coating of the foam backings used.

Test Methods

Maximum elastic strength, rupture elongation

The measurement is conducted according to DIN 53 504 with standard test samples of size S 2 at a separation speed of 300 mm/min.

Elasticity Modulus

The elasticity modulus is determined analogous to ASTM D 882 with following test sample dimensions: Test strip length = 140 mm; mounting length (distance between clamp brackets) = 100 mm; test sample width = 15 mm. Speed of separation = 25.4 mm/min.

Rocking shear strength

The adhesive film of dimensions 50 mm x 20 mm, which is to be tested and equipped on one end on both sides with a non-tacky handle region (obtained by laminating a 25 pm strong biaxially stretched polyester film of dimensions 20 mm * 13 mm (Hostaphan RN 25)), is glued to the middle of a highly polished, square steel plate of dimensions 40 mm * 40 mm * 3 mm (length * width * thickness) to determine the rocking shear strength. The steel plate is equipped in the middle on the back side with a 10 cm long steel pin which is seated vertically on the plate surface. The test sample obtained is glued with a force of 100 N to the adhesive surface to be tested (time of contact pressure = 5 sec), and rested for 5 min in unloaded conditions. After applying the chosen rocking shear load by applying a weight (lever arm and mass of the weight can be chosen), the time to failure of the adhesive joint is determined.

Strip force

An adhesive film of dimensions 50 mm x 20 mm (length x width) is glued with the upper end of the non-tacky handle region (see above) between two steel plates (arranged to be aligned with each other) of the dimensions 50 mm x 30 mm as described according to the rocking shear strength durability test, but with a mounting pressure of 500 N each. At the bottom end of the steel hook the steel plates feature another steel plate by which the measurement fixtures can be attached for the measurement at the lower clamping bracket of the tensile tester. The adhesive joint is stored for 24 h at + 40 °C. After reconditioning to RT the adhesive film strip is removed at a strip speed of 1000 mm/min parallel to the

plain of adhesion. The required strip force is measured during the stripping in N/cm. The steel plates are afterwards examined for the presence of residue from the adhesive material.

Adhesion Surface on Glass

Adhesive film strips of dimensions 20 mm x 50 mm are glued centered to the straight steel substrate of dimensions 200 mm x 100 mm. The composite produced this way is placed vertically and congruently on a glass plate of the same dimensions and pressed against it at the center with 100 N. The time pressure is applied is 5 sec. The measurements are taken threefold. The adhesion surface obtained on the glass plate is determined visually and given in percent of the adhesive film surface.

Adhesion Surface on Rough Fiber Wallpaper

Adhesive film strips of dimensions 20 mm x 50 mm are glued centered to the straight steel substrate of dimensions 200 mm x 100 mm to determine the adhesive surface area on rough substrates. The composite produced this way is placed vertically and congruently on a painted rough fiber wallpaper of the same size (wallpaper: Erfurt corn size 52; paint: Herbol Zenit LG; wall paper glued to particle board) which is powdered thinly with aluminum bronze, and pressed against it evenly in the center at 100 N. The time pressure is applied is 5 sec. The measurement is taken threefold. The samples can easily be removed vertically from the powdered rough fiber wallpaper. The adhesive surface area obtained is visually determined by the amount of aluminum bronze transferred to the adhesive film surface and given in percent of the adhesive film surface.

Test for Residue Free and Destruction Free Release

A corresponding test is conducted within the scope of determining the strip force (see above) for the substrates steel // steel. To also determine a residue free and destruction free release on other substrates, for example PMMA // painted rough fiber wallpaper (wallpaper: Erfurt corn size 52; paint: Herbol Zenit LG; wall paper glued to particle board), corresponding test samples are produced as described above in "Strip Force", and the adhesive joint is released either by machine or manually (stripped). The presence of residue of adhesive materials on the glued substrates and the destruction of the adhesive substrates are determined.

Examples

Example 1

An adhesive material consisting of 20 parts SPS block-copolymer (Vector 8508, Exxon), 80 parts SIS block-copolymer (Vector 4211, Exxon), 100 parts of a penta-ester of partially hydrated colophony (pine resin; Foralyn 110, Hercules), and 1 part of a primary antioxidant (Irganox 1010, Ciba Geigy) {= Recipe 1], or an adhesive material consisting of 50 parts natural rubber with a K-value of 145, 50 parts Foralyn 110, and 1 part Irganox 1010 [= Recipe 2] is applied by cold lamination on both sides of the foam backings listed below. The chosen foam material is placed on adhesive material present on siliconized release paper and firmly rolled afterwards five times with a rubber coated steel roll of 25 cm in width at an application pressure of 50 N. The intermediate product obtained this way is coated in identical manner on the second side with adhesive material. Tests are conducted of the samples obtained this way after 24 hours of conditioning in a climate controlled chamber (50% relative humidity, T = RT = 23 °C). Test samples (adhesive films) are punched out in all cases in cross direction to the production direction of the foam backing used. Following properties are obtained:

Sample name	**Foam Trade name**	**Foam Type**	**Manufacturer**
3.001A	Alveolit TEE 0400.35	EVAc copolymer	Alveo AG
3.006	Alveolit TEE 0500.51	EVAc copolymer	Alveo AG
3.009	Alveolit TEE 0400.35	EVAc copolymer	Alveo AG
3.010	Alveolit TEE 0500.51	EVAc copolymer	Alveo AG
3.000A	-	-	-
3.000B	-	-	-

Sample name	**Foam thickness in μm**	**Volume weight in kg/m^3**	**Applied adhesive mass Side A // B**	**Recipe**
3.001A	350	250	200 // 210 g/m^2	[1]
3.006	510	200	200 // 200 g/m^2	[1]
3.009	350	250	56 // 56 g/m^2	[2]
3.010	510	200	56 // 56 g/m^2	[2]
3.000A	-	-	360 g/m^2	[1]
3.000B	-	-	650 g/m^2	[1]

Sample name	Highest elast. force -length	Rupture elongation	Strip force length/cross	Foam rupture ?	Residue and rupture free stripped?
3.001A	39 N/cm	1050%	15/n.d. N/cm	No	Yes
3.006	30 N/cm	950%	15/13 N/cm	No	Yes
3.009	-	-	- N/cm	Yes	No
3.010	-	-	- N/cm	Yes	No
3.000A	44 N/cm	1300%	- N/cm	-	Yes
3.000B	59 N/cm	1200%	- N/cm	-	Yes

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Sample name	Rocking shear durability	E-modulus foam backing -length/cross	Adhesive area in % on Rough wallpaper // Glass	
3.001A	> 30 days	14.3 MPa / 9.1 MPa	about 90%	about 90%
3.006	> 30 days	10.0 MPa / 6.7 MPa	> 95%	> 95%
3.009	n.d.	14.3 MPa / 9.1 MPa	n.d.	n.d.
3.010	n.d.	10.0 MPa / 6.7 MPa	n.d.	n.d.
3.000A	4-6 days	- -	about 30%	40-50 %
3.000B	8-12 days**	- -	about 40%	40-50 %

n.d. = not determined
** **Rough fiber wallpaper splits**

In the given example only those adhesive films containing a intermediate foam backing can be released free of residue and damage, which utilize adhesive materials with a sufficiently high rupture strength. An almost 100 percent adhesive surface area is achieved both on the chosen rough fiber substrate as well as on the smooth glass substrate by utilizing the intermediate foam backing. The adhesive strength (rocking shear durability) obtained for the adhesive films utilizing the intermediate foam backing is clearly above that of adhesive films which don't have an intermediate foam backing.

Example 2

An adhesive material according to Recipe [2] is applied on both sides to the foam materials listed below. The production of the adhesive samples occurs analogous to Example 1. Following results are obtained:

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Sample name	Foam Trade name	Foam Type	Manufacturer
3.006	Alveolit TEE 0500.51	EVAc copolymer	Alveo AG
3.019	Alveolit TEE 0501.2	EVAc copolymer	Alveo AG

Sample name	Foam thickness in µm	Volume weight in kg/m^3	Applied adhesive mass Side A // B	Recipe
3.006	510	200	200 // 200 g/m^2	[1]
3.019	1200	200	200 // 200 g/m^2	[1]

Sample name	Highest elast. force	Rupture elongation	Strip force cross	Foam rupture ?	Residue and rupture free stripped?*
3.006	30 N/cm	950%	13.3 N/cm	No	Yes
3.019	40 N/cm	1100%	20.5 N/cm	No	Yes

* **Adhesive joint substrates steel // steel**

The release forces have been considerably reduced in the current example by utilizing a very thin foam which make the release markedly easier.

Patent Claims

1. Adhesive tape for an adhesive joint which can be released by stripping residue free and without damage, with a foam backing, which is coated on one or both sides with a self-adhering material, **characterized by**

a) having a self-adhering adhesive material applied to at least one of the two sides of the foam backing, whose ratio of rupture force to strip force (removal force) at a strip angle of less than 10° to the adhesive area is larger than 1.2 : 1, and
b) the foam backing featuring an elasticity modulus below 16 MPa and a thickness of 200 - 600 µm.

2. Adhesive tape according to Claim 1, characterized by the foam backing being coated on both sides with a tacky adhesive material.

3. Adhesive tape according to Claim 1, characterized by the tacky adhesive material

being one on the basis of block-copolymers, especially those containing vinyl-aromatic components.

4. Adhesive tape according to Claim 1, characterized by the tacky adhesive material being one on the basis of block-copolymers containing polymer blocks of vinyl-aromates (A-block) and those formed by polymerization of 1,3 diene (D-blocks).

5. Adhesive tape according to Claim 1, characterized by the tacky adhesive material containing tackifiers and possibly additional mixture components and / or additives.

6. Adhesive tape according to Claim 1, characterized by the foam backing having an E-modulus of 4 - 15 MPa.

7. Adhesive tape according to Claim 1, characterized by the foam backing having a thickness of 300 - 550 µm.

8. Adhesive tape according to Claim 1, characterized by it being present in form of sections, whereby the one end of the section features a non-tacky handle and the other end an adhesive surface possibly diminishing towards the other end.

9. Adhesive tape according to Claim 8, characterized by the handle being formed by laminated film sections which sides touching the tacky adhesive material are equipped to be anti-adhesive.

10. Use of a section of an adhesive tape according to one of the claims 1-9 for a residue and damage free adhesive joint which can be released, characterized by pulling on one end of the section.

11. Use according to Claim 10, characterized by using a section coated on both sides with tacky adhesive material together with a hook, a base plate or an object to be mounted, and possibly being prefabricated onto the latter.